

## Aminomethylierte Dihydroxybenzole

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### Abstract of DE4429344

Oxidation hair dyes contain aminomethylated dihydroxybenzenes as coupler components together with usual developer components.

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The invention concerns oxidation coloring means for coloring human hair, which contains usual developer components and as Kupplerkomponenten of aminomethylsubstituierte Dihydroxybenzolderivate.

For coloring hair the so-called oxidation hair coloring means play a preferential role because of their intensive colors and good authenticity characteristics. Such hair coloring means contain oxidation coloring material before products in a cosmetic carrier. As oxidation coloring material before products developer components and Kupplerkomponenten are used. The developer components train the actual coloring materials under the influence of oxidizing agents or of atmospheric oxygen among themselves or under clutch with one or more Kupplerkomponenten.

As developer components primary aromatic amines with a further, in para or ortho position free or substituted hydraulic XY or amino group, Diaminopyridinderivate present, hetero-cyclic Hydrazone, 4-Aminopyrazolonderivat as well as 2,4,5,6-Tetraaminopyrimidin and its derivatives are usually used.

Special representatives are for example p-Toluylendiamin, 2,4.5.6 - Tetraaminopyrimidin, p-aminophenol, N, N-Until (2-hydroxyethyl) - p-phenylendiamin, 2 (2,5-Diaminophenyl) - ethanol, 2 (2,5-Diaminophenoxy) - ethanol, 1-Phenyl-3-carboxyamido-4-amino-pyrazolon-5 and 4-Amino-3 methyl phenol, 2-Hydroxy-4,5,6-triaminopyrimidin, 2,4-Dihydroxy-5,6-diaminopyrimidin and 2,5,6-Triaminohydroxypyrimidin.

As Kupplerkomponenten m-Phenylendiaminderivate, Naphthole, Resorcin and Resorcinderivate, Pyrazolone and m-Aminophenole are usually used. As Kupplersubstanzen in special alpha - Naphthol, Pyrogallol, 1,5, 2,7 - and 1,7-Dihydroxynaphthalin, 5 - amine CO<sub>2</sub> methyl phenol, m-aminophenol, Resorcin, Resorcinnomonomethylether, m-Phenylendiamin, 1-Phenyl-3-methyl-pyrazolon-5,2,4-Dich 3-aminophenol, 1,3-Bis (2,4-diaminophenoxy) - are suitable propane, 2-Chlor-resorcin, 2-Chlor-6-methyl-3-aminophenol and 2-Methylresorcin.

A certain developer component can form also very different color SCN by combination with different Kupplern. Nevertheless often does not succeed, with the help of only one developer component to the variety of natural hair color nuances comes. In practice therefore usually a combination of different developer components and Kupplerkomponenten is necessary, in order to receive a only one, naturally working hair colouring. There is constantly therefore need of new, improved Kuppler/developer combinations.

It is task of the instant invention to find new Kupplerkomponenten which fulfill to oxidation coloring material before products to requirements placing in particular.

Good oxidation coloring material before products must fulfill primarily the following conditions: They must train the desired color nuances with the oxidative clutch in sufficient intensity and authenticity. Furthermore they must possess a good absorptive power on human hair, whereby no noticeable differences between strained and freshly regenerated hair may exist (equalizing abilities). They should be steady against light, warmth the influence of chemical reducing agents, z. B. against continuous waving liquids. Finally they are not to anfärben the scalp too much, and above all they are to be

▲ top harmless in toxicological and dermatologischer regard.

The European disclosure writing EP 0,398,702 a2 reveals hair coloring means, which contain aminomethylsubstituierte m-Aminophenole as Kuppler.

Article of the instant invention oxidation hair coloring means are containing for coloring Keratinfasern, in particular human hair, Kupplerkomponenten and developer components in an aqueous carrier, by the fact characterized that at least a usual developer component in a quantity from 0,01 to 20 Gew. - %, preferably 0.5 to 5 Gew. - %, and as Kupplerkomponente a Dihydroxybenzolderivat of the formula I

EMI3.1

or its salt in a quantity from 0,01 to 20 Gew. - %, preferably 0.5 to 5 Gew. - % contained %, in each case related to the entire oxidation hair coloring means, whereby X stands for a hydrogen atom, a halogen atom or a C1-C4-Alkylgruppe, and R<1> and R<2> independently hydrogen, C1-C4-Alkylgruppe or C2-C4-Hydroxyalkylgruppen mean and the unit NR<1> R<2> also hetero-cyclic to represent can do a not aromatic, if necessary an oxygen or a nitrogen atom containing, five or Sechs-Ring.

Particularly N, N-Until (beta - hydroxyethyl) are suitable - 2,4-dioxybenzylamin or N, N-Until (beta - hydroxyethyl) - 2,4-dioxy-3-methylbenzylamin as Kupplerkomponenten.

The Dihydroxybenzolderivat of the formula I is partly literature well-known and leaves itself according to well-known synthesis methods, z. B. by Aminomethylierung after Mannich, (details see example part), partly are new they manufacture in addition, like z. B. the N, N-Until (beta - hydroxyethyl) - 2,4-dioxy-3-methylbenzylamin; an exact description of its synthesis takes place in the example part.

As usual according to invention developer components applicable in the oxidation hair coloring means more near already

above the substances described are applicable. The developer and Kupplerkomponenten know thereby both and free bases and in form i of inorganic or organic salts, z. B. the hydrochlorides or hydraulic bromide, to be used.

The coloring means according to invention supply a broad spectrum of application technology interesting oxidation colors in the range yellow-brown to dark-brown nuances of high brilliance, good equalizing ability and good authenticity characteristics.

Preferential coloring means supply nuances in the red range; such nuances can be obtained with the oxidation hair coloring means according to invention in particular if the developer component 2.4.5.6 - Tetraamino pyrimidin is.

In a preferable embodiment the hair coloring means according to invention contain additionally usual directpulling coloring materials, z of the formula I and the developer components for the further modification of the color nuances beside the Dihydroxybenzolderivaten. B. from the group of the Nitrophenylen diamine, Nitroaminophenole, Anthrachinone or Indophenole, like z. B. under the international designation and/or. Trade name HC Yellow 2, HC Yellow 4, methyl-yellow, fluorine-yellow, basic Yellow 57, Disperse orange 3, HC talk 3, HC talk BN, cardinal-red, red Y, red X, basic talk 76, HC Blue 2, Nitroblau, Disperse Blue 3, basis Blue 99, HC Violet 1, Disperse Violet 1, Disperse Violet 4, Disperse Black 9, basis Brown 16, Pikraminsäure and Rodol 9 R, well-known connections, in a quantity from 0,01 to 20 Gew. - %, related to entire oxidation hair coloring means.

It is not necessary that the Dihydroxybenzolderivate of the formula I, which represents developer components or the fakultativ contained directpulling coloring materials uniform in each case connections. Rather also mixtures of different Dihydroxybenzolderivate of the formula I, developer components and directpulling coloring materials can be used into the hair coloring memo according to invention. In particular can do beside the Dihydroxybenzolderivaten de Formel I further usual, above mentioned. Kupplerkomponenten contained its.

In the hair coloring means according to invention developer components and Kupplerkomponenten are used to each other generally in approximately molecular quantities. Even if the molecular employment proved as appropriate, then a certain surplus of individual oxidation coloring material before products is not unfavorable, so that developer components and Kupplerkomponenten in a molar ratio vo 1: 0,5 to 1: 2 to be contained can.

For the production of the coloring means according to invention the oxidation coloring material before products in a suitable aqueous carrier are trained. Such carriers are z. B. Creams, emulsions, gels or also tensidhaltige foaming solutions, z. B. Shampoos, foam aerosols or other preparing, which are suitable for application on the hair.

The aqueous carrier contains usually net and emulsifying agents like anionische, nichtionische or ampholytische Tenside, z. B. Fat alcohol sulfates, Alkansulfonate, alpha - Olefinsulfonate, Fettalkoholpolyglykolethersulfate, alkyl glycosides, products of accumulation of ethyl oxide to Fettalkohole, at fatty acids, at alkyl phenol, at Sorbitanfettsäureester, at Fettsäurepartialglyceride and Fettsäurealkanolamide; Thickening agent, z. B. Fettalkohole, fatty acid of paraffin oils, fatty acid esters and other fat components in emulsified form; water-soluble polymere thickening agent like natürl. Gummen, z. B. Gum arabicum, Karaya gum, Guar gum, Johannsbrotkernmehl, Leinsamengummen; biosynthetic Gummen, z. B. Xanthan gum and Dextrane; synthetic Gummen, z. B. Agar agar and Algin, strength parliamentary groups and derivatives such as amyl eye, Amylopektin and Dextrine; modified cellulose molecules, z. B. Methylcellulose, hydroxyalkyl cellulose and Carboxymethylcellulose; Clay/tone such as z. B. Bentonit or full-synthetic hydraulic colloids, z. B. Polyvinyl alcohol or Polyvinylpyrrolidon; hair-maintaining additives, like z. B. water-soluble kationische polymers, anionische polymers, nichtionische polymers, amphotere or zwitterionische polymers, Pantothensäure, Vitamine, plant extracts or Cholesterin; pH control agent, complexing agent and perfume oils as well as reducing agents for the stabilization of the contents contents of, z. B. Ascorbic acid; finally also Farbsto can be contained for dyeing the cosmetic preparing. In particular Johannsbrotkernmehl and Guarkernmehl are as ideal thickening agents for non-pourable woods, z. B. Sandelholz, containing nature hair coloring materials admits. These thickening agents are preferential corresponding using, if the oxidation hair coloring means according to invention contain additionally non-pourable woods.

The ingredients of the aqueous carrier are used for the production of the hair coloring means according to invention into quantities usual for this purpose; z. B. become emulsifying agents in concentrations from 0,5 to 30 Gew. - % and thickening agents in concentrations from 0,1 to 25 thread. - % of the entire coloring means assigned.

The oxidative development of the colouring can take place in principle with atmospheric oxygen. Preferred however a chemical oxidizing agent is used, particularly if beside the colouring a lightening effect at the hair is desired. As oxidizing agents come in particular hydrogen peroxide or its accumulation products at urea, Melamin or sodium borate as well as mixtures from such products of accumulation of hydrogen peroxide with Kaliumperoxidisulfat into consideration.

The preparation of the oxidizing agent is appropriately mixed directly before the hair coloring with the preparation from the oxidation coloring material before products. The ready for use hair coloring preparation developing thereby should prefers pH-incoming goods within the range of 6 to 10 exhibiting. The application of the hair coloring means is particularly preferential in a weakly alkaline environment. The application temperatures can lie in a range between 15 and 40 DEG C. After an induction period of approx. 30 minutes the hair coloring means is removed by rinsing out from the hair which can be colored. Washing afterwards with a shampoo is void, if a strongly tensidhaltiger carrier, z. B. a coloring shampoo was used.

The following examples are to describe the invention article more near, without limiting it however on that.

#### Examples

##### 1. Manufacture examples

1. Literature quotation for N, N-Until (beta - hydroxyethyl) - 2,4-dioxybenzylamin x HCl (c1) ?leather? (1950), 82

2. Synthesis of N, N-Until (beta - hydroxyethyl) - 2,4-dioxy-3-methyl-benzylamin x HCl (K2).

6.2 g (0.05 mol) 2-Methylresorcin were solved with 45 DEG C in 50 ml 50%iger hydrochloric acid. Then 5.85 g (0.05 mol) were course-dripped 3 - beta - Hydroxyethyl oxazolidin (DOS 2 24 636). After 1 grant with 45 DEG C the product one sucked off, with ethanol one washed afterwards and i.V. with 60 DEG C dried. White crystals: 166 DEG C.

#### Sample applications

Hair coloring means according to invention in form of a hair coloring cream emulsion in the following composition were manufactured

```
< tb> < TABLE> Columns=2>
< tb> Head Col 1: Fettalkohol C12-C14
< tb> Head Col 2: 2.0 g,
< tb> SubHead Col 1: Fettalkohol C12-C20
< tb> SubHead Col 2: 8.5 g,
< tb>
< tb> SubHead Col 3: Fettalkohol C12-C14 + 2 EO sulfate, sodium salt 28%ig
< tb> SubHead Col 4: 20 g,
< tb>
< tb> SubHead Col 5: Water
< tb> SubHead Col 6: 40 g,
< tb>
< tb> SubHead Col 7: Developer component (E1-E7)
< tb> SubHead Col 8: 0.0075 mol,
< tb>
< tb> SubHead Col 9: Kupplerkomponente (K1-K2)
< tb> SubHead Col 10: 0.0075 mol,
< tb>
< tb> SubHead Col 11: Na2SO3 (inhibitor)
< tb> SubHead Col 12: 1.0 g,
< tb>
< tb> SubHead Col 13: (NH4) 2SO4
< tb> SubHead Col 14: 1.0 g,
< tb>
< tb> SubHead Col 15: concentrated ammonia solution
< tb> SubHead Col 16: to pH = 10,
< tb>
< tb> SubHead Col 17: Water
< tb> SubHead Col 18: ad 100 G.
< tb> < /TABLE>
```

The ingredients were mixed in sequence with one another. After addition of the oxidation coloring material before products and the inhibitor first the pH value of the emulsion on 10 adjusted became with concentrated ammonia solution, then with water to 100 g one filled up.

The oxidative development of the colouring became with 3%iger hydrogen peroxide solution as oxidation solution conducted. For this wur 100 g of the emulsion with 50 g hydrogen peroxide solution (3%ig) offset and mixes.

The coloring cream became on approx. 5 cm long Strähnen standardized, to 90% did not turn grey however particularly pre-treated people hair laid on and there 30 to minutes with 27 DEG C left. After completion of the dyeing process the hair purged was washed, with a usual Haarwaschmittel and dried afterwards.

As developer components E1 to E7 became  
E1: P-Toluylendiamin,  
E2: P-aminophenol,  
E3: 2,4,5,6-Tetraaminopyrimidin,  
E4: 2-Dimethylamino-4,5,6-triaminopyrimidin,  
E5: 2-Morpholino-4,5,6-triaminopyrimidin,  
E6: 2-Piperidyl-4,5,6-triaminopyrimidin,  
E7: 2-Methylamino-4,5,6-triaminopyrimidin  
begun.

Table 1  
EMI10.1